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Leather Research and Technology at the National Bureau of Standards

A Review and Bibliography

UNITED STATES DEPARTMENT OF COMMERCE

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A summary of the activities of the Leather Section Organic and Fibrous Materials Division, of the National Bureau of Standards is given. Some of the more noteworthy accomplishments mentioned are: Establishment of the optimmum pH of 3.0, below which leather is not stable during storage; method for the quantitative determination of amino acids in collagen; measurement of the size and distribution of pores in leather; methods of impregnating leather with polymers; measurement of the physical constants of leather and other polymers; investigation of the tanning properties of synthetic organic compounds; and the construction and design of special equipment for laboratory evaluation of performance.

A list of publications by members of the National Bureau of Standards Leather Section staff pertaining to collagen, leather, and other polymers is included.

I. Introduction

The basic leather-forming constituent of animal skins is the white connective tissue, which consists primarily of a complex protein called collagen. Leather is collagen whose resistance to hydrolysis and putrefaction has been increased by the action of tanning agents.

The two types of materials most commonly used for tanning are the natural tannins found in the bark, wood, leaves, and fruits of certain trees and shrubs, and chromium salts. Other tanning agents used are iron, aluminum, zirconium salts, fish oils, formaldehyde, sulfite cellulose waste liquors, and synthetic organic compounds.

Leather has certain inherent properties, such as high resistance to stitch tear, good dimensional stability, water vapor permeability, and moldability, that make it highly desirable for the manufacture of shoes, gloves, and other items of clothing. Some physical properties of leather, such as tensile strength, flexibility, softness, abrasive resistance, and water absorption, can be controlled to a high degree by using skins of various types and origins and selecting a tanning and finishing process suitable to the end product.

The application of scientific principles to the tanning of hides was not generally accepted until about the beginning of the twentieth century. Most of the early research on leather by industrial and Government scientists was devoted to measuring physical properties, standardizing test procedures, and improving the serviceability of leather, particularly for footwear.

A broad program of leather research and development, of interest to Government agencies and private industry, was initiated at the National Bureau of Standards prior to World War I. The increased volume and importance of this work was recognized March 1, 1926, by the formation of the National Bureau of Standards Leather Section.

A knowledge of the structure of collagen, the leather-forming protein of hides and skins, and of the mechanism of tanning, is of direct industrial importance to the tanners, the shoe manufacturers, and all who are called on to appraise the properties of leather. The Bureau recognized the need for more basic information and has directed a large portion of the research work of the Leather Section toward fundamental studies of collagen, concepts of the mechanism of tanning, and measurement of the physical and chemical properties of collagen and leather. The staff of the Leather Section has closely cooperated with the American Leather Chemists' Association as active members, officers on the Council, and members of committees, and it has published more than 80 research papers in the Journal of the American Leather Chemists' Association.

Other functions of the Leather Section are to advise and assist Government agencies on problems related to the procurement of leather items and the identification and examination of articles made, in whole or in part, of leather. The Section also furnishes general information to the public on leather and miscellaneous leather products.

International recognition of the work of the Bureau's Leather Section was achieved in 1937 [3.17][1] when the National Bureau of Standards

[1] Figures in brackets indicate the literature references at the end of this paper.

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was requested to prepare a report on the deterioration of leather by acid for inclusion in a German treatise on the subject. Members of the staff have participated in meetings of the International Union of Leather Chemists' Societies and visited laboratories in England and on the European continent. Foreign students from Brazil, China, India, Indochina, Jordan, Mexico, and Turkey, sponsored by their respective governments, have been assigned to the Bureau to work on leather problems and study the organization and operation of the leather laboratory.

II. Leather Research and Technology

The experimental work of the Leather Section may be classified under four categories: Properties, development, serviceability, and test methods. The first of these relates to basic studies designed to investigate the physical and chemical properties of leather and other polymeric materials. The data accumulated in the course of this work have both a practical and a theoretical value. The second category pertains to technological research and development, and has resulted in the development of new processes for tanning, finishing, and treatments of leather for specific uses. The third category concerns the stability and serviceability of leather, and consists of a study of the mechanism of degradation and factors influencing serviceability. The fourth category, test methods, has resulted in the development of laboratory methods for the physical and performance characteristics of leather and chemical methods for analyzing leather and leathermaking materials. The following section gives a brief synopsis of the research work under each subject. The numbers in brackets refer to the pertinent publications classified under the subjects listed in section III.

1. Chemical and Physical Properties of Leather and Other Polymeric Materials

a. Physicochemical

Extensive studies of physical and chemical properties of collagen and leather have been carried out at the National Bureau of Standards. As a result of the complexity of the chemical and physical structure of the basic material, a combination of both chemical and physical studies is carried out in some individual researches. This results in a large class of investigations on properties which cannot be classed as either purely chemical or purely physical, but as a combination of the two, which may be termed physicochemical.

Studies that can be classed as physicochemical involve in most instances interactions of leather or collagen with water in some form. The strong adsorptive capacity of leather and collagen for water and water vapor and the importance of this adsorbed moisture on the properties of the leather has led to a series of studies on the adsorption and its variation with temperature, tannage, etc. [1.13, 1.20]. Extensions of these studies are being made to study heats of wetting and the effect of various factors on the values obtained on leather and other fibrous polymers of interest (see fig. 1). The related factor of transfer of water vapor from a region of high humidity to one of lower humidity, at which leather excels, has been extensively investigated and led to theories concerning the mechanism of this transfer [1.21]. Studies of the rate of shrinkage [1.17] are another example of investigations on the role of the water-leather interaction. Extensive measurements have been made on the swelling of leather, collagen, and modified collagen in water and various solutions of interest [1.43].

The physical constants of fibers are of less immediate practical importance, but are of great value in understanding the complex nature of collagen and the processes involved in converting collagen to leather. In general, studies of fiber constants are made under varying conditions of tannage, moisture content, temperature, composition, etc., so that considerable information is obtained in such researches.

For thermodynamic characterization of a material, data are required for density [1.11], expansivity [1.16], compressibility [1.25], and specific heat. The latter three quantities and their variations with temperature are of prime importance in understanding and predicting the behavior of any material. These constants all have been and are continually being studied at the Bureau and, as expected in such measurements, interesting phenomena are uncovered that are being investigated further. Expansivity measurements, for example, uncovered phenomena leading to studies of rates of shrinkage with resulting data on heats and entropies of activation [1.17]. Compressibility measurements at the high pressures required for solids involve techniques and equipment not widely understood. Following designs perfected in other laboratories, equipment has been assembled and used to measure compressibilities, etc. [1.24]. The scarcity of such equipment and the importance of compressibility data have led to studies being conducted on other synthetic organic polymers of interest [1.25, 1.35] (see fig. 2). A number of other investigations on materials of interest to other divisions of the Bureau [1.34, 1.36, 1.40] and the Geophysical Laboratory of the Carnegie Institution have been made [1.42]. Data on specific heats are now being accumulated in the course of basic studies on interactions of moisture and leather.

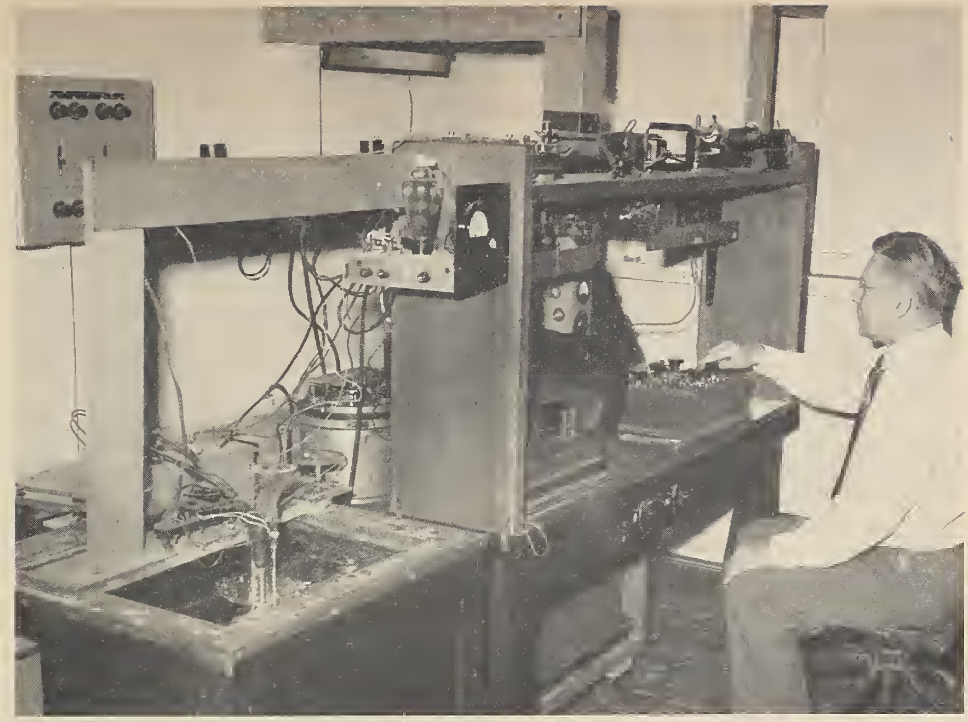

FIGURE 1. *Calorimeter.*

Calorimetric equipment in the Mineral Products Division used for determining the heat of wetting and specific heat of collagen and leather.

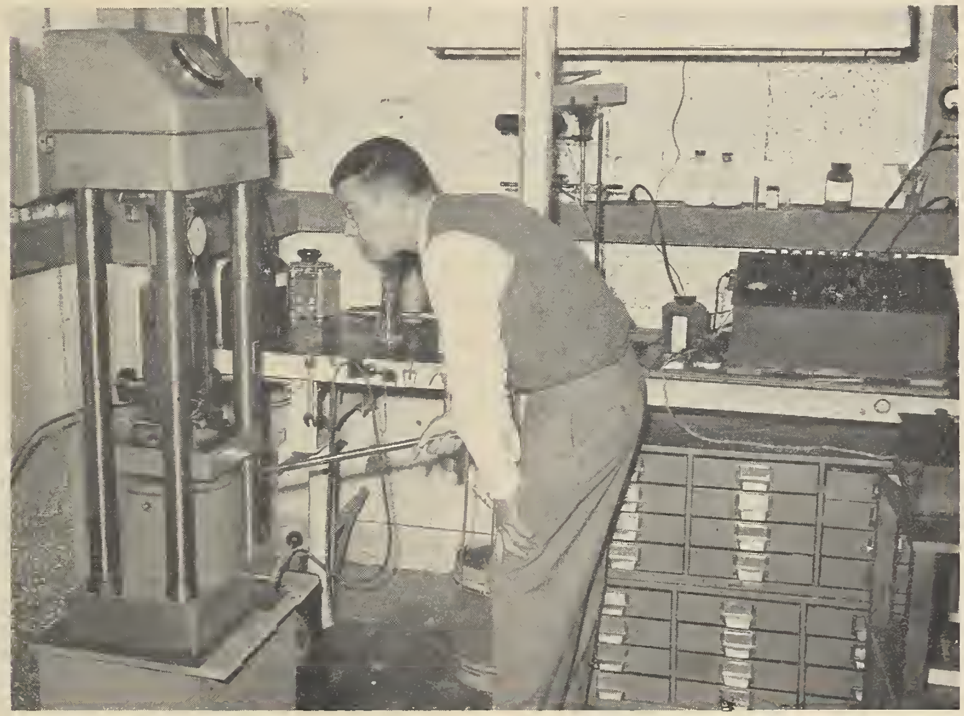

FIGURE 2. *Compressibility measurements at high pressures on leather and other polymers being carried out in the Bureau's Leather Section.*

The specimen is immersed in liquid in the massive vessel in the press. A leakproof piston forced into the vessel by the press generates pressure by compressing the liquid and thereby subjects the specimen to hydrostatic pressure. Motion of the piston is followed by the dial gage and the pressure by the change of resistance of a coil of resistance wire mounted inside the vessel. All polymers studied yield compressibilities intermediate between those of crystalline solids and true liquids with natural fibrous polymers being less compressible than the synthetics.

b. Physical

The purely physical properties or constants of a fibrous material, such as leather, consist in general of two distinct quantities: One, which characterizes the material consisting of fibers plus voids, may be called the matrix value; the second, which pertains to the fibers alone, may be called the fiber value.

The matrix values are obviously of immediate practical value to the user or purchaser of leather products, as these will largely govern the behavior of leather in service. A number of studies of such properties have been carried out and designed to cover most practical situations in which leather has been used or use has been contemplated. Such studies cover for example the following subjects: Mechanical properties, such as tensile strength, stretch, tearing strength, stiffness, bursting strength, rigidity, and flexural resistance [1.1, 1.29]; thermal properties involving studies of shrinkage temperature, area stability, thermal conductivity [1.9, 1.13], etc.; electrical properties of resistance and dielectric constant [1.28], etc.; and structural properties such as pore-size distribution in the fibers and leather.

The determination of the pore-size distribution over a wide range of pore diameters by pressure porosimeter techniques has produced fundamental information concerning the physical and chemical structure and characteristics of the matrix of leather and collagen fibers, as well as the relative amounts of void spaces available for absorption of tannins, impregnants, transfer of water vapor, etc. [1.46]. Electron microscopy used to supplement this study gave information concerning the geometry of the fibrils. In figure 3 (left), the spaces between lines of fibril contact are seen to be possible pore sites. In addition to these pores, there is a possibility of pores existing between the units of subfibrillar structure that are suggested by figure 3 (right).

c. Chemical

Skin collagen, being a natural fibrous protein, is a highly complex polymer that occludes extraneous materials, such as fats, salts, and other proteins. Fundamental chemical studies of collagen have required investigation of methods of purification to remove extraneous materials and permit preparation of a chemically reproducible purified collagen [1.18].

The measurement of the electrophoretic properties of purified collagen [1.19], the changes induced by relatively simple chemical reactions, such as esterification, deamination, and various tanning procedures, determinations of the combining weights [1.7] and the reactive basic groups, the study of the amino acid structure by column and

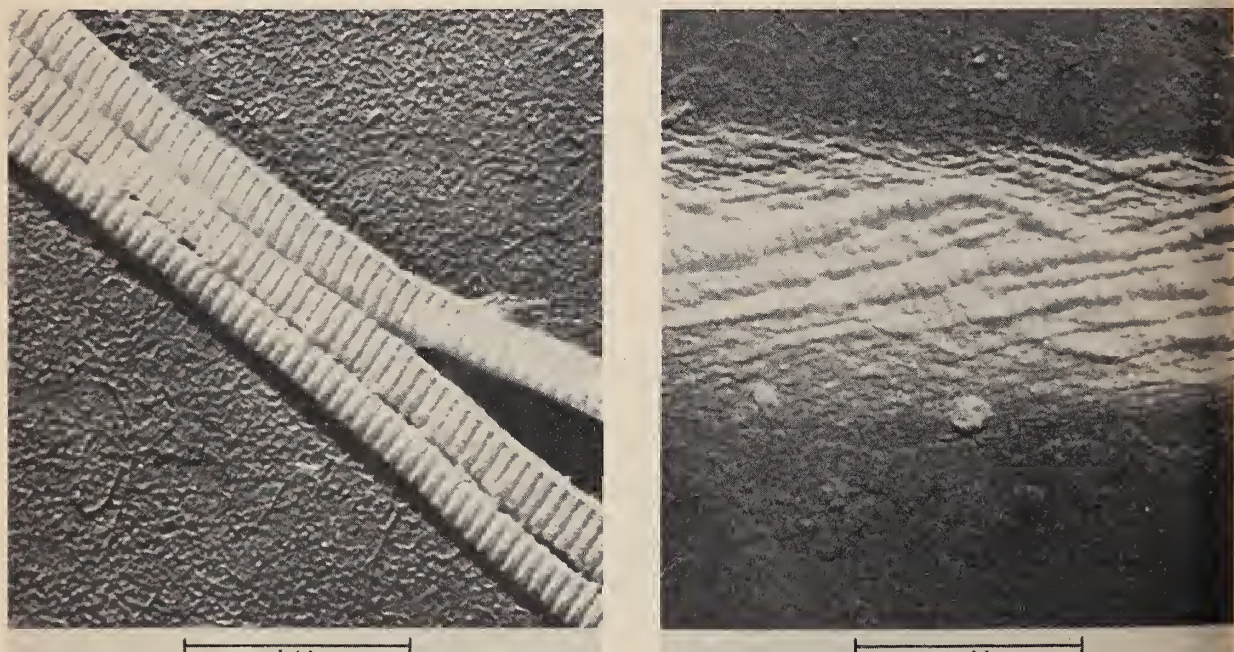

FIGURE 3. *Study of the fine structure of collagen.*

Left, Electron micrograph of three collagen fibrils obtained from kangaroo tail tendon showing the periodic cross-striations of a typical fibril. Palladium shadowed 4 : 1. Right, Electron micrograph of a single collagen fibril obtained from kangaroo tail tendon showing the substructure of a swollen fibril. Palladium shadowed 4 : 1.

FIGURE 4. *Mobility measurement of collagen particles using horizontal electrophoretic cell.*

The Abramson electrophoresis cell permits the mobility of collagen particles to be determined in aqueous media at various pH values. The speed with which the particles traverse a known distance in the eye piece micrometer of the microscope is determined. The resistance unit on the right permits constant control of the field strength. From a plot of mobility versus pH, the isoelectric point (pH of electrical neutrality) of the material is determined.

paper chromatographic techniques [1.37], and the amide nitrogen content [1.38] of collagen and hide powder have produced fundamental data that should be of great value in the determination of the structure of collagen and the mechanism of tanning (see fig. 4).

2. Technological Research and Development

a. Tanning Materials

In 1924 a survey by the U. S. Department of Commerce stated that 40 percent of the vegetable tanning materials consumed in the United States in 1922 were imported. Further, it was pointed out that 99 percent of the chrome ore, used in the United States at that time, was imported. The potential value of a substitute tanning material to the national economy was recognized, and as a result the Bureau initiated research work on synthetic tanning materials.

Representative syntans of various types were prepared from information available at that time and were evaluated by tanning tests. The results were published in a series of papers [2.7 through 2.10] and helped lay the foundation for the development of present day satisfactory syntans by industry. An investigation of the tanning properties of sulfite cellulose [2.11], a byproduct of the paper industry, demonstrated that this material could be used in conjunction with vegetable tanning extracts. Later, during World War II, it was shown that iron could be substituted for chromium during an emergency [2.14].

b. Oil Treatments

During World War II, leather and tanning materials were on the strategic list. The Army Quartermaster Corps, recognizing the need for conservation, sponsored a research project at the Bureau on improving the stability and serviceability of leather and the development of test methods for military items. An oil treatment was soon developed, which when applied to sole leather gave a significant increase in wear [2.15]. The War Production Board recommended this treatment for all civilian sole leather.

c. Impregnated Leather

Research on improving the serviceability of leather was continued after the war and a more recent development, sponsored by the Department of the Navy, in which leather is impregnated with polymers has resulted in substantial improvement in the wearing quality and water resistance of sole leather [2.18, 2.20] (see fig. 5). Leather, because of the tightly woven condition of the natural fibers of which it is composed, acts under certain conditions as an efficient filter, and this property limits the materials that may be used as impregnants. Early attempts to use certain solutions of commercial polymers as impregnants were not successful, and research work was directed towards impregnating the leather with

FIGURE 5.—*Service tests of sole leather.*

Soles, cut from the same location on opposite sides of a hide, one side of which is finished as commercial sole leather, are attached to shoes and worn until a small hole appears in one sole. The relative wear is calculated from the original thickness. The pair of soles pictured is typical of such a test. The sole on the bottom was cut from a commercial sole leather tannage, the one on the top was cut from a side of leather lightly tanned and impregnated with rubber.

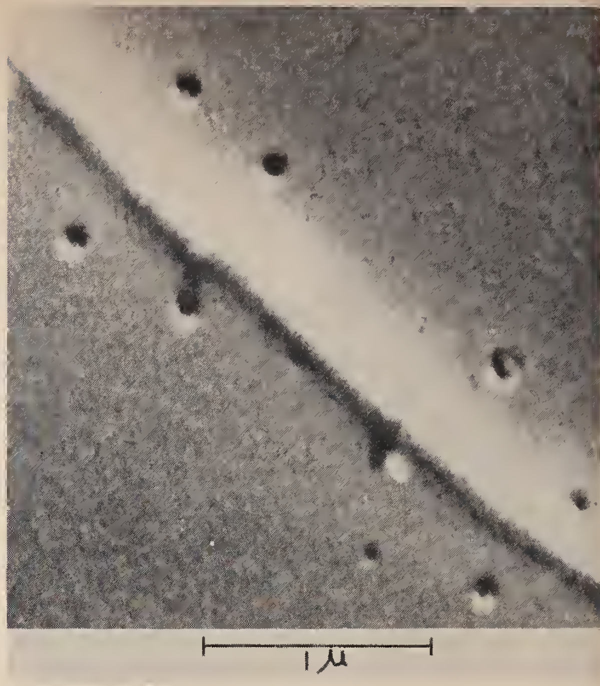

FIGURE 6. *Leather impregnation studies by electron microscopy.*

Left, Electron micrograph of a single collagen fibril obtained from the center split of tanned crust leather. Chromium shadowed 4 : 1. Right, Electron micrograph of a single collagen fibril obtained from the center split of tanned crust leather after impregnation with natural rubber. Chromium shadowed 4 : 1.

monomers and polymerization in situ [2.18]. As a result of other research work on the pore-size distribution in leather and particle-size distribution in rubber latex [1.39], it became apparent that leather could be impregnated by soaking the crust leather (tanned, but not finished) in solutions of specially prepared or selected commercial polymers [2.23]. The extent of impregnation of the leather matrix with solutions of rubber was studied with the aid of electron microscopy.

The characteristic collagen striations of the fibril persist even after tanning, and figure 6 (left), shows the sharp periphery and cross striations observed in shadowed fibrils of leather. On impregnation with rubber, the distinguishing features of the fibrils are hidden by a rubber film, as shown by the shadowed fibril of figure 6 (right). The holes observed in the background of figure 6 (right), are probably formed in the preparation of the specimen for electron microscopy and are further indications of a rubber sheath.

3. Stability and Serviceability

a. Deterioration

The deterioration of leather and its useful life is a function of its environment, usage, and tannage. Investigations of the mechanism of degradation have shown that highly ionizable acids, whether added in the tanning or finishing processes of leather manufacture or absorbed from the surrounding atmosphere, are a major factor in deterioration.

The Bureau made a thorough study of the effect of acid on leather and published a series of research papers on the subject. Some of the results of major importance of this investigation were the development of a standard procedure for determining acidity [4.6] and the determination of the optimum pH, below which leather could not be expected to maintain its properties during prolonged storage [3.8]. Oxygen, moisture, temperature, and the catalytic effect of traces of copper and iron salts in the leather are contributing factors in deterioration and have been the subject of research [3.10, 3.19, 3.22].

b. Fungusproofing Studies

Prior to World War II mildew on leather was considered of relatively minor importance and little effort was made to prevent its growth. However, the military forces stationed in tropical areas soon found that the growth of mildew on numerous items of equipage was a serious problem. As a result, the Army sponsored a project at the Bureau for the development of fungicidal treatments and test methods.

These studies showed that the principal effect of mildew on leather, other than appearance and psychological effect on the observer, was the removal

Figure 7. *Mildewproofing of leather.*

The pair of service boots pictured was stored under tropical conditions for several months. Before storage, the boot which is free from mildew was treated with a fungicidal formulation developed at this Bureau. The other boot, covered with mildew, had received no mildewproofing treatment.

of greases, causing stiffness and loss in strength [3.25, 3.27]. As a result of this investigation, a specification for the fungicidal treatment of leather was prepared [5.33] and quantitative methods for the determination of fungicides in leather were developed [5.21, 4.26] (see fig. 7).

4. Test Methods

The development of physical and chemical test methods and testing equipment for leather, leather products, and leathermaking materials to be used in procurement specifications and in evaluation and development work has been a major phase of the work on leather at the Bureau (see figs. 8 and 9).

The accurate determination of moisture in leather has long been a serious problem to analysts, as the results for all other chemical constituents are expressed on the oven-dry basis. NBS research [4.11] showed that control of the atmospheric humidity in the drying oven would permit moisture determinations to be made with a precision indicated by an average deviation of a single observation of 0.03 percent. This method has been adopted as the ultimate standard in Federal Specifications [5.21].

Because the properties of leather vary considerably over the area of a hide, it is important that the sample for test represent as nearly as possible the average for the hide [4.18]. A particularly noteworthy contribution was made to the sampling of side upper leather. The selection of the sampling location was based on the correlation between the value obtained for a specific test from a specific location and the average for that test in the corresponding side. The most suitable location on a hide for sampling, for all tests required in acceptance testing, was determined.

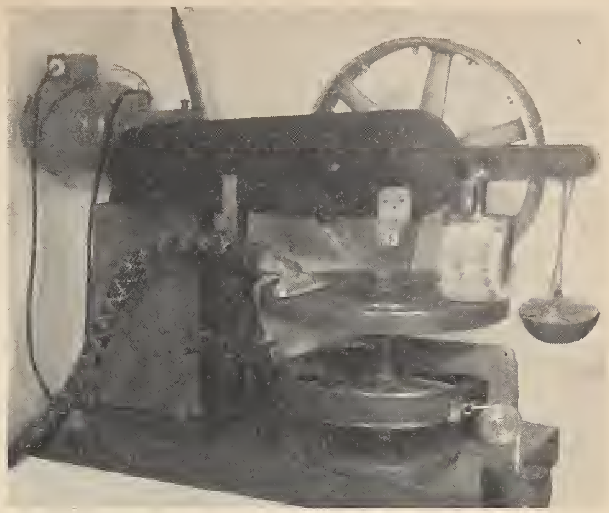

Figure 8. *Abrasion machine.*

The variety of conditions to which shoes are exposed in use, such as temperature, moisture, and walkway surfaces, precludes the use of a single laboratory test to determine performance. Resistance to abrasion is one factor in evaluating serviceability and is used to determine the relative value of new products and treatments.

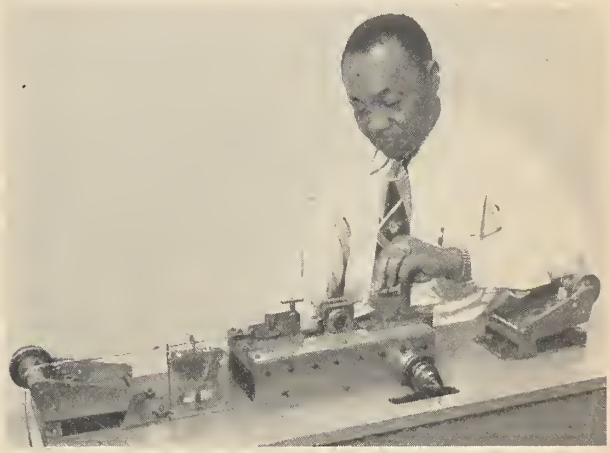

Figure 9. *Flex-tension machine.*

The flexibility of leather is not appreciably affected by extreme temperature changes such as may be encountered in service, and its resistance to flexural fatigue under normal conditions of use is very high. To evaluate this property in a reasonable time, the breakdown is accelerated by repeated bending of the specimen around rollers at a predetermined angle of bend and tension. The machine pictured is one of several that have been designed in the Leather Section of the National Bureau of Standards to evaluate performance characteristics of leather.

Performance requirements are often more indicative of the serviceability of a product than chemical or physical properties. Some of the test methods and apparatus which have been developed under this category include abrasive resistance of heavy leather [4.8], flex life [4.27], accelerated aging [3.22], luggage performance [2.19] water penetration [1.14], and compressibility [1.12].

5. Federal Specifications

The First Federal Specification for leather, United States Government Specification for Leather Belting, was adopted by the Federal Specifications Board, July 1922, as Standard Specification No. 37 and was printed, October 1923, as National Bureau of Standards Circular 148. Since that time, 36 specifications for leather and leather products have been promulgated. These specifications were prepared by the Leather and Leather Products Committee, Federal Specifications Board.

The Federal Specifications Board has recently been abolished and an assigned agency system for the preparation of Federal Specifications adopted by the General Services Administration. The Department of Commerce has been assigned the responsibility for the preparation of most of the Federal Specifications for leather and leather products and the National Bureau of Standards has been designated as the preparing activity responsible for their development. Normally, coordination of Federal Specifications for leather with industry and the Government is on an individual basis. However, if the Bureau desires group assistance, advisory groups may be set up for a particular specification or similar specifications, with the concurrence of the General Services Administration.

Copies of Federal Specifications and the Index of Federal Specifications and Standards may be obtained upon application accompanied by check, money order, cash, or Government Printing Office coupons, to the General Services Administration, Business Service Center, Region 3, Seventh and D Streets, S. W., Washington 25, D. C. That office will also honor deposit account numbers issued by the Government Printing Office. Prices may be obtained from the Index of Federal Specifications and Standards or from the General Services Administration Regional Offices. When ordering Federal Specifications, the classification symbol and the title of the specification should be stated. Single copies of product specifications required for bidding purposes are available without charge at the General Services Administration Regional Offices in Atlanta, Boston, Chicago, Dallas, Denver, Kansas City, Mo., Los Angeles, New York, San Francisco, Seattle, and Washington, D. C. A list of Federal Specifications for leather and leather products is given in section III.

III. Bibliography

The National Bureau of Standards publications listed in sections 1 to 4, inclusive, and 6 have appeared in the Bureau regular series or in the scientific and technical journals cited. A list of Federal Specifications for leather and leather products is given in section 5.

For papers and publications not printed by the Government, the name of the journal or of the organization publishing the article is given in abbreviated form, with the volume number, page, and year of publication, in the order named. Information regarding their availability and prices can be obtained only from the publisher or organization sponsoring the publication. Reprints of these articles are not available from the Government Printing Office. Those marked with an asterisk may be secured without charge by addressing the Leather Section, National Bureau of Standards, Washington 25, D. C.

Publications marked (OP) are out of print and are consequently no longer available. They may in general be consulted in technical and public libraries. Where the prices of Government publications are given, they may be purchased from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. Single copies of Research Papers, beginning with number 2399, are not available for sale, but the Superintendent of Documents will reprint 100 or more copies, and request for the purchase price should be mailed promptly to that Office.

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IV. Other Government Sources of Information

Several other agencies of the Federal Government issue publications on leather. While these are too numerous to be listed here, the following sources may be indicated:

DEPARTMENT OF COMMERCE

Office of Technical Services

Commercial Standards establish standard quality requirements, methods of test, rating, certification, and labeling of commodities, and provide uniform bases for fair competition. They are developed by voluntary cooperation among manufacturers, distributors, consumers, and other interests, upon the initiative of any of these groups, through a regular procedure of the Commodity Standards Division. The procedure is explained in a pamphlet entitled "Voluntary Standards Adopted by the Trade," obtainable free of charge from the Commodity Standards Division, Office of Technical Services, Department of Commerce, Washington 25, D. C. A list of Commercial Standards is given in Commercial Standards Classified List, Revised to February 1, 1955, Catalog No. 978.

Simplified Practice Recommendations. These publications relate to the reduction of excessive variety of manufactured products, or of methods. Simplified Practice Recommendations are developed by voluntary cooperation among manufacturers, distributors, consumers, and other interests, upon the initiative of any of these groups, through a regular procedure of the Commodity Standards Division. A list of recommendations is given in Simplified Practice Recommendations, Alphabetical List Revised to February 1, 1954, Catalog No. 979.

Copies of the following pertinent Simplified Practice Recommendations or Commercial Standards may be consulted in public and college libraries, or may be purchased from the Superintendent of Documents, United States Government Printing Office, Washington 25, D. C., at the prices indicated.

Luggage (trunks and suitcases), Simplified Practice Recommendation R215–46 (5¢).

Materials for safety wearing apparel (second edition), Commercial Standard CS129–47 (5¢).

Work gloves (with supplement), Commercial Standard CS139–47 (10¢).

Bureau of the Census

Census of Manufacturers: Volume I, General Summary (1949), Volume II, Statistics by Industry (1949), Volume III, Statistics by States (1949).

Bureau of Foreign Commerce

Foreign Commerce Weekly, Trade Promotion Series. Published weekly. (Information on leather and related products; exports, imports, foreign markets, international trade.)

Office of Technical Services

Newsletter. Published monthly. (Notice of Publications Board and foreign technical reports, their price and availability; includes leather in some issues.)

Office of Business Economics

Survey of Current Business. Published monthly. (Includes statistics related to the leather industry in some issues.)

Business and Defense Services Administration; Leather, Shoes, and Allied Products Division

The Administration handles all commercial problems for foreign and domestic relating to production, distribution, and sales of leather, shoes, and allied products.

Higher World Production and Consumption of Leather Footwear, December 1953. (Business Information Service.) (Available from the Superintendent of Documents, price 10¢.)

FEDERAL TRADE COMMISSION

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DEPARTMENT OF THE AIR FORCE

Index of Specifications and Bulletins approved for U. S. Air Force Procurement, October 1, 1953. Issued every 6 months. (Headquarters Air Materiel Command, Wright-Patterson Air Force Base, Dayton, Ohio. Issued only to authorized persons.)

DEPARTMENT OF THE ARMY

Index of Specifications. (Includes MIL and JAN Specifications.) October 1, 1953. Issued every 6 months. (For sale by the Superintendent of Documents, Government Printing Office, Washington 25, D. C.)

DEPARTMENT OF THE NAVY

Index of Specifications used by the Navy. Navsanda publication No. 62. Issued quarterly. (Copies of the Index, Part I and unclassified specifications and standards listed therein, and all military specifications in 5000 and 6000 series on leather may be obtained from the Bureau of Supplies and Accounts, Department of the Navy, Washington 25, D. C.)

DEPARTMENT OF AGRICULTURE

Eastern Utilization Research Laboratory, Philadelphia 18, Pa.

Issues research papers on hides, tanning materials, and leather; Farmers' Bulletins on Shoes, Home Tanning, and others. A list of publications and methods of obtaining them may be secured from the Laboratory.

A complete list of Government periodicals for which subscriptions are taken is published by the Superintendent of Documents, Government Printing Office, Price List 36, 76th edition, November 1954, and is obtainable gratis from that office.

Washington, April 15, 1955.

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DEPARTMENT OF COMMERCE

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